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भारतीय मानक

बिटुमन और एन्थ्रासाइट कोयले के पेट्रोग्राफिक विश्लेषण की पद्धतियाँ

भाग 3 मिसरल समूह संघटन ज्ञात करने की पद्धति (पहला पुनरीक्षण)

Indian Standard METHODS FOR THE PETROGRAPHIC ANALYSIS OF BITUMINOUS COAL AND ANTHRACITE

PART 3 METHOD OF DETERMINING MACERAL GROUP COMPOSITION

(First Revision)

ICS 74.040

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BUREAU OF INDIAN STANDARDS MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002

NATIONAL FOREWORD

International Standard

This Indian Standard (Part 3) (First Revision) which is identical with ISO 7404-3: 1994 'Methods for the petrographic analysis of bituminous coal and anthracite — Part 3: Method of determining maceral group composition' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of Solid Mineral Fuels Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

This standard was published in 1979 which was largely based on ISO/DIS 7404-3. ISO/DIS 7404-3 has subsequently been published in 1984 and revised in 1994 which is now available as ISO 7404-3:1994 'Methods for the petrographic analysis of bituminous coal and anthracite — Part 3: Method of determining maceral group composition'. The Committee, therefore, decided to revise this standard to completely align it with ISO 7404-3:1994 and publish as a dual number standard. Consequently, the title has been modified as 'Methods for the petrographic analysis of bituminous coal and anthracite: Part 3 Method of determining maceral group composition'.

The text of ISO Standard has been approved as suitable for publication as Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

- a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.
- b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their place are listed below along with their degree of equivalence for the editions indicated. However, that International Standard cross-referred in this adopted ISO standard, which has subsequently been revised, position in respect of latest ISO standard has been given:

Corresponding Indian Standard

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ISO 7404-1: 1994 Methods for the petrographic analysis of bituminous coal and anthracite — Part 1: Vocabulary	IS 9127 (Part 1): 1992 Methods for the petrographic analysis of coal: Part 1 Definition of terms relating to petrographic analysis of coal (first revision)	Technically equivalent with minor deviations
ISO 7404-2: 1985 Methods for the petrographic analysis of bituminous coal and anthracite — Part 2: Method of preparing coal samples	IS 9127 (Part 2): 2002 Methods for the petrographic analysis of bituminous coal and anthracite: Part 2 Method of preparing coal samples (first revision)	Identical
ISO 7404-4: 1988 Methods for the petrographic analysis of bituminous coal and anthracite — Part 4: Method of determining microlithotype, carbominerite and minerite composition	IS 9127 (Part 4): 2001 Methods for the petrographic analysis of bituminous coal and anthracite: Part 4 Method of determining microlithotype, carbominerite and minerite composition	do

Degree of Equivalence

IS 9127 (Part 3): 2002

ISO 7404-3: 1994

Indian Standard

METHODS FOR THE PETROGRAPHIC ANALYSIS OF BITUMINOUS COAL AND ANTHRACITE

PART 3 METHOD OF DETERMINING MACERAL GROUP COMPOSITION

(First Revision)

1 Scope

This part of ISO 7404 specifies a method of determining the proportions of the maceral groups (and the minerals if desired) in coals. It is concerned only with determinations made on polished particulate blocks using reflected white light. If needed, the proportions of the individual macerals may be determined by the same procedure. It is not concerned with the determination of the proportions of naturally occurring maceral associations (i.e. microlithotypes, see ISO 7404-4).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 7404. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 7404 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO

maintain registers of currently valid International Standards.

ISO 7404-1:1994, Methods for the petrographic analysis of bituminous coal and anthracite — Part 1: Vocabulary.

ISO 7404-2:1985, Methods for the petrographic analysis of bituminous coal and anthracite — Part 2: Method of preparing coal samples.

ISO 7404-4:1988, Methods for the petrographic analysis of bituminous coal and anthracite — Part 4: Method of determining microlithotype, carbominerite and minerite composition.

ISO 7404-5:1994, Methods for the petrographic analysis of bituminous coal and anthracite — Part 5: Method of determining microscopically the reflectance of vitrinite.

3 Definitions

For the purposes of this part of ISO 7404, the definitions given in ISO 7404-1 apply.

4 Principle

A representative sample of coal is used to prepare a particulate block as described in ISO 7404-2. This is examined using a reflected light microscope and the maceral groups are identified under an immersion medium by their relative reflectance, colour, size and morphology. Their proportions are determined by a point count procedure.

5 Material

Immersion medium, having a suitable refractive index and compatible with the microscope objective.

NOTE 1 It is recommended that an oil with a refractive index of 1,518 0 as specified in ISO 7404-5 be used, especially if the reflectance of the macerals is to be measured.

6 Apparatus

- **6.1 Reflected light microscope**, having an immersion objective of magnification between \times 25 and \times 60 and an eyepiece of magnification between \times 8 and \times 12. The eyepiece incorporates a fine crossline graticule.
- 6.2 Mechanical stage, capable of advancing the specimen laterally by equal steps of such length that only a negligibly small proportion of the particles examined receives more than one count on the same particle. The step length is equal to half the maximum particle diameter, i.e. 0,5 mm to 0,6 mm for samples with a standard top particle size of 1 mm. The stage also permits a similar stepped advance in the perpendicular direction. The lateral movement is actuated preferably by the counter mechanism, whereas the perpendicular movement may be satisfactorily performed manually.
- **6.3 Counter**, capable of registering the counts in each category and preferably the grand total of petrographic components.
- **6.4 Sample mounting equipment**, comprising slides, modelling clay and levelling device.

7 Preparation of coal sample

Prepare and polish a particulate block as described in ISO 7404-2.

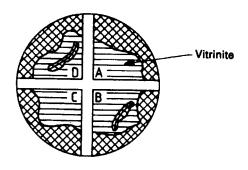
8 Procedure

Adjust the microscope (6.1) for Köhler illumination. Set up the levelled particulate block on the stage, place the immersion medium (clause 5) on the surface of the block, focus and observe the image in the microscope. Identify the material lying under the intersection of the crosslines and carry out the point count procedure as follows.

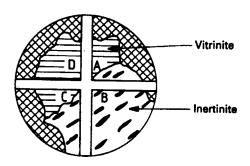
Crosslines on	Action
Vitrinite (V), liptinite (L) or inertinite (I)	Operate the counter for appropriate maceral group.
Mounting medium	Ignore the point.
Minerals (M)	Operate the counter for pyrite, shale, etc. or ignore the point (see the last three paragraphs of the introduction and clause 10).
Boundary between macerals or between maceral and mounting medium	Examine in turn the material ly- ing immediately adjacent to crossline intersection in the top right, bottom right, bottom left and top left quadrants. Take the first of these which does not have a boundary in it, and oper- ate the counter for this material (see figure 1).
Empty pore in a maceral or a void	Ignore the point

Advance the block by one step in the left-to-right direction, and continue counting and traversing the specimen. At the end of a traverse, advance the block by a step of at least equal length in the perpendicular direction to start the next parallel traverse. Choose the step length to ensure a uniform counting of points over the surface of the block.

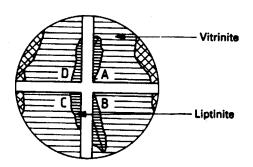
Count a total of at least 500 points.



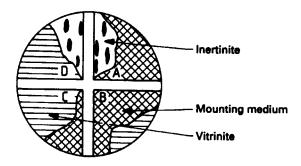
a) Normal case - count point A (vitrinite)



b) Boundary case — count point B (inertinite)



c) Boundary case - count point C (liptinite)



d) Boundary case - the point is not counted

NOTE — Width of crosslines exaggerated for clarity.

Figure 1 — Normal and boundary cases between macerals or between maceral and mounting medium

9 Expression of results

Calculate the volume percentage of each component [vitrinite (V), liptinite (L), inertinite (I)], which is equal to the percentage number of points counted on it, expressing the results to the nearest integer. The form of the results depends on the procedure adopted with regard to minerals (see the last three paragraphs of the introduction) and is expressed on one of the following bases:

- a) minerals ignored:% V + % L + % I = 100
- b) minerals (M) counted:% V + % L + % I + % M = 100
- c) minerals matter (MM) calculated: % V + % L + % I + % MM = 100

In alternative c), minerals are ignored in the point count but the volume percentage of mineral matter is calculated from the determined ash by means of an accepted empirical relationship.

The procedure adopted and the number of points counted shall be indicated in the test report.

NOTE 2 Examples of equations to calculate the mineral matter, % MM, as a volume percentage, are as follows:

$$% MM = 0.61w_A - 0.21$$
 ...(1)

% MM =
$$\frac{w_{\rm M}}{2.07 - 0.011w_{\rm A}}$$
 ...(2)

where

w_A is the ash content, as a mass percentage on the air-dried basis;

 w_{M} is the mineral matter content, as a mass percentage, given by the equation

$$w_{\rm M} = 1.08w_{\rm A} + 0.55w_{\rm S}$$

where $w_{\rm S}$ is the sulfur content, as a mass percentage on the air-dried basis.

Equation (2) is based on assumed relative densities of 1,35 and 2,8 for the macerals and mineral matter respectively.

These equations have been found satisfactory in certain coal basins but may not necessarily apply globally. It is essential for the user to establish suitable equations for the coals being analysed.

10 Precision

10.1 Repeatability limit

The repeatability limit of the determination of the volume percentage of a component is the value of the difference between two single determinations, each based on the same number of points counted, carried out by the same operator on the same block using the same apparatus, below which 95 % of such differences are expected to lie. The repeatability limit may be calculated from the formula

$$\left(2\sqrt{2}\right)\sigma_{t}$$

where σ_t is the theoretical standard deviation.

Provided that the operator makes negligible errors in classifying the macerals, the results of an analysis are subject to standard deviations calculable on the basis of the binominal distribution.

Where p % of the total number of points counted, N, is registered for a given maceral group, the theoretical standard deviation, σ_t , of p is given by the equation

$$\sigma_{\rm t} = \sqrt{\frac{p(100-p)}{N}}$$

Values based on counts of 500 points for the theoretical standard deviation, coefficient of variation and repeatability calculated for a range of volume percentages of a component are given in table 1.

Table 1 — Theoretical standard deviation and repeatability limit of the percentage of a component, based on counts of 500 points

Volume %,	Standard deviation, $\sigma_{\rm t}$	Coefficient of variation,	Repeatability limit, $\left(2\sqrt{2}\right)\sigma_1$
5	1,0	20	2,8
20	1,8	9	5,1
50	2,2	4,4	6,3
80	1,8	2,3	5,1
95	1,0	1,1	2,8

NOTE 3 For example, if the volume percentage of vitrinite in a sample is 80 %, then an operator can expect to obtain two results differing by less than 5,1 percentage points (e.g. 78 % and 83 %) in 19 cases out of 20.

10.2 Reproducibility limit

The reproducibility limit of the determination of the volume percentage of a component is that value of the difference between two single determinations, each based on the same number of points counted, carried out by two different operators on two different subsamples taken from the same sample, using different equipment, below which 95 % of such differences are expected to lie. The reproducibility limit is given by the formula

$$\left(2\sqrt{2}\right)\sigma_{\circ}$$

where σ_0 is the observed standard deviation.

Values of the observed standard deviation normally exceed the values for the theoretical standard deviation given in table 1 owing to the misidentification of the macerals by different operators and to variation between subsamples; they have been found to vary from approximately 1,5 to 2,0 times the theoretical values, depending on the rank and the heterogeneity of the coal.

11 Test report

The test report shall include the following information:

- a) a reference to this part of ISO 7404;
- all details necessary for identification of the sample;
- c) the name and address of the testing laboratory;
- d) date of test;
- e) the number of points counted;
- f) whether minerals were counted or ignored, or whether the mineral matter was calculated, and (if calculated) the equation used;
- g) the results obtained;
- h) any other characteristics of the sample observed during the analysis that may be relevant to the use of the results.

NOTE 4 It may also be useful to include nominal magnification, point count stage step size and line traverse spacing.

Annex A

(informative)

Bibliography

[1] International Handbook of Coal Petrography, published by the International Committee for Coal and Organic Petrology (ICCP).

NOTE 5 The second edition (1963), together with the first supplement (1971, corrected and revised in 1985), second

supplement (1975) and third supplement (1993) may be obtained from Professor D.G. Murchison, Fossil Fuels and Environmental Geochemistry, Drummond Building, University of Newcastle, Newcastle-upon-Tyne, NE1 7RU, United Kingdom.

International Standard

ISO 7404-5: 1994 Methods for the petrographic analysis of bituminous coal and anthracite — Part 5: Method of determining microscopically the reflectance of vitrinite

Corresponding Indian Standard

IS 9127 (Part 5): 1986 Methods for the petrographic analysis of coal: Part 5 Microscopical determination of the reflectance of vitrinite

Degree of Equivalence

Technically not equivalent. However, IS 9127 (Part 5) is being considered for revision to align it with ISO 7404-5: 1994

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2: 1960 'Rules for rounding off numerical values (revised)'.

Bureau of Indian Standards

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Review of Indian Standards

Amend No.

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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